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PRECIPITATED SILICA GRANULES
[Fällungskieselsäuregranulate]

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Description

[0001] This invention relates to precipitated silica granules, a process for their production, and their use.

[0002] It is known to produce precipitated silica granules with the help of a nozzle atomizer or an atomizer for two substances, by using a suspension of precipitated silica with a pH of at least 4 and a solid content greater than 18 wt% (EP-B 0 018 866).

[0003] The known precipitated silica granules, which are intended as a reinforcing filler for elastomers, have the disadvantage of having a high abrasion and, thus, an undesiredly high dust content. For this reason, they are not particularly suitable for other applications, in which the absence of dust is important.

[0004] Moreover, the known precipitation silica granules are unsuitable as a carrier silica for choline chloride solution, if active ingredient adsorbates having an active ingredient content of 50 wt% are to be produced.

[0005] Thus, the task is to produce precipitated silica granules with a low dust content and with a good absorptive capacity for choline chloride solution.

[0006] The subject matter of the present invention is precipitated silica granules that are characterized by the following dusting parameters:

maximum dust ≤ 5.0 wt%

* claim and paragraph numbers correspond to those in the foreign text.

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dust number ≤ 10.0 wt%
settling number to 5% ≤ 5 s
settling number to 2% ≤ 50 s

[0007] The precipitated silica granules of this invention can be prepared by adjusting a suspension of precipitated silica to a pH of 2 to 3.9 and using spray drying. The spray drying can be carried out by means of a one-component nozzle in a spray tower or using the FSD method. The suspension of precipitated silica can have a solid content of 17 to 19.5 wt%.

[0008] The precipitated silica granules of this invention can be used for adsorbing liquid agents, in particular vitamins, for example vitamin E acetates, and for adsorbing choline chloride solution.

Product A

[0009] In one embodiment of the invention, the precipitated silica granules can be characterized by the following dusting parameters:

maximum dust ≤ 1.0 wt%
dust number ≤ 2.0 wt%
settling number to 5% ≤ 1.0 s
settling number to 2% ≤ 2.0 s

[0010] These precipitated silica granules can also be characterized by the following physicochemical characteristics:

tapped bulk density	g/l	263 – 278
BET surface	m^2/g	199 – 203
CTAB surface	m^2/g	178 – 180
DBP number	g/100 g	220 – 320
average particle size d$_{50}$	μm	314 – 480
pH		4,9 – 5,2

[0011] The precipitated silica granules of this invention can also have the following physicochemical characteristics:

conductivity: 200 to 1,200 μS
moisture content: 3 to 8%
Alpine sieve residue (63 μm): # 100%

SEM (= scanning electron microscope) images as in Figure 1

[0012] The precipitated silica granules of this invention can be prepared by first precipitating the silica in a conventional manner in accordance with DE-A 14 67 019, then filtering it. The filter cake of precipitation silica can be further processed to produce the sprayable suspension of precipitated silica as in DE-B 24 47 613.

[0013] In a preferred embodiment, the suspension of precipitated silica can be adjusted to a pH of 2.0 to 3.9.

[0014] This suspension of precipitated silica can be spray dried using a one-component nozzle in a spray tower to form the precipitated silica granules of this invention.

[0015] Die one-component nozzle and the spray tower are known devices for spraying a solid containing suspensions. They are described in K. Masters, Spray Drying Handbook, Third Edition, George

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Godwin Limited, London / John Wiley and Sons, New York (1979), pp. 208 to 231.

[0016] In an additional embodiment of the invention, the suspension of precipitated silica can be sprayed to produce the precipitated silica granules of this invention using the FSD method.

[0017] The FSD drying or granulation technique was developed in 1980 by the firm NIRO in Copenhagen. Basically, this method involves a combination of spray drying and fluidized bed drying.

[0018] The drying air produced in a gas burner enters centrally through the top of the tower and leaves the drying chamber, also through the top of the tower, in a cyclone or dust filter. The spray drying chamber comprises a slightly conical top, a short cylinder, and a narrow cone with an integrated, stationary fluidized bed at the bottom of the cone.

[0019] The product to be spray dried is typically atomized by one or more pressure nozzles and carried downward in co-current flow with the drying air. The liquid atomizer is placed centrally in the top air diffuser. Due to the strong fluidization in the fluidized bed and the recirculation of the fine particles entrained by the exhaust air in the drying chamber, the spray drying occurs in a turbulent cloud of powder. At the same time, the fines separated out in the dust filter are immediately returned pneumatically into the atomization cloud of the pressure nozzle. The required residual moisture content and the particle distribution are achieved by the

mode of operation of the liquid atomizer and the integrated fluidized bed or additional downstream vibration fluidized beds. The degree of agglomeration and, thus, the particle size distribution, can be influenced by various operating parameters over a relatively broad range.

[0020] A product of this method is shown in Figure 5, using SEM (= scanning electron microscope) images.

Product B

[0021] In another embodiment of the invention, the precipitated silica granules can be characterized by the following dusting parameters:

maximum dust	≤ 5.0 wt%
dust number	≤ 10.0 wt%
settling number to 5%	≤ 5 s
settling number to 2%	≤ 50 s

[0022] These precipitated silica granules can also be characterized by the following physicochemical characteristics:

tapped bulk density	g/l	204 – 208
BET surface	m^2/g	423 – 430
CTAB surface	m^2/g	337 – 340
DBP number (cont.)	g/100 g	220 – 320
average particle size d_{50}	μm	332 – 400
pH		3,8 – 4,0

[0023] The precipitated silica granules of this invention can also have the following physicochemical characteristics:

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conductivity:                          200 to 1,200 μS
moisture content:                      3 to 8%
Alpine sieve residue (63 μm):          # 100%
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SEM (= scanning electron microscope) images as in Figure 3

[0024] The precipitated silica granules of this invention can be prepared by first precipitating the silica in a conventional manner in accordance with DE-A 31 44 299, then filtering it. The filter cake of precipitation silica can be further processed to produce the sprayable suspension of precipitated silica as in DE-B 24 47 613.

[0025] In a preferred embodiment of the invention, the suspension of precipitated silica can be adjusted to a pH of 2.0 to 3.9. This suspension of precipitated silica can be spray dried using a one-component nozzle in a spray tower to form the precipitated silica granules of this invention.

Product C

[0026] In another embodiment of the invention, the precipitated silica granules can be characterized by the following dusting parameters:

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maximum dust                    ≤ 5.0 wt%
dust number                     ≤ 10.0 wt%
settling number to 5%           ≤ 5 s
settling number to 2%           ≤ 50 s
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[0027] These precipitated silica granules can also be characterized by the following physicochemical characteristics:

tapped weight	g/l	278 – 294
BET surface	m^2/g	180 – 190
CTAB surface	m^2/g	168 – 171
DBP number	g/100 g	220 – 320
average particle size d_{50}	μm	306 – 321
pH		4,0 – 4,1

[0028] The precipitated silica granules of this invention can also have the following physicochemical characteristics:

conductivity: 200 to 1,200 μS
moisture content: 3 to 8 wt%
Alpine sieve residue (63 μm): # 100%

SEM (= scanning electron microscope) images as in Figure 4

[0029] The precipitated silica granules of this invention can be prepared by first precipitating the silica in a conventional manner in accordance with DE-A 31 44 299, then filtering it. The filter cake of precipitation silica can be further processed to produce the sprayable suspension of precipitated silica as in DE-B 24 47 613.

[0030] In a preferred embodiment of the invention, the suspension of precipitated silica can be adjusted to a pH of 2.0 to 3.9. This suspension of precipitated silica can be spray dried using a one-component nozzle in a spray tower to form the precipitated silica granules of this invention.

[0031] The precipitated silica granules of this invention have the advantage compared to known precipitated silica granules of a significantly lower dust content. Consequently, they can be used quite well for purposes that require a low amount of dust. They also

have a good adsorption capacity for agents such as choline chloride solution, and vitamins, such as vitamin E acetate.

Example 1

Preparation of the precipitated silica granules

Product A

[0032] The precipitation silica is prepared as in DE-A 14 67 019, example 1.

[0033] The process is as follows:

[0034] 11.5 liters water at 80°C is placed in an 80-liter container and dilute water glass solution is added until the pH is 10. Then, at 86°C and over a period of 100 minutes, a sodium silicate solution with a specific gravity of 1.063 and a sulfuric acid solution having 90 g H_2SO_4/liter are fed into the receiving vessel at the same time. The feed rate of the acid is approximately 1/3 to 1/4 that of the alkali silicate solution. A pH between approximately 10 and 11.5 is maintained. The addition continues for about 100 minutes. During this time, the viscosity increases from 2.2 units to 11.5 units. It then drops to 3.8 units. The addition of acid and water glass is continued for around 100 minutes, while keeping the pH in the above-mentioned range. The precipitate solution contains approximately 50 g SiO_2/liter. It is then acidified to a pH of 7 and then to a pH of 2.5.

[0035] The precipitation silica that is obtained is filtered off. The precipitation silica filter cake is washed and transferred

into a suspension of precipitated silica, as in example 1 of DE-C 24 47 613. The process is as follows:

[0036] 260 kg/min precipitation silica filter cake with a water content of 82% is fed in by means of the twin screw at 295 rpm through the constricted discharge to increase the kneading/mixing effect at the same time as 0.6 liter/min dilute, aqueous 30% sulfuric acid from the line, into the 1.5 m^3 dissolver container. During this process, the acid concentration is kept such that the suspension of precipitated silica has a pH of 3. The dissolver container has a diameter of 1.2 m, a height of 1.5 m, and is cylindrical in shape. The dissolver, which is a double disk dissolver, has a diameter of both disks of 0.4 m and is driven by an electric motor, which has a power of 36.7 kW (50 HP). The rotational speed of the two disks is 21 m/s (1,000 rpm). The submersible probes are used to keep the filling level between 0.8 and 1.20 m, so that 1.5 kg suspension of precipitated silica is in the dissolver container per unit of 1 kg precipitation silica filter cake. The suspension of precipitated silica is removed from the zone of greatest turbulence using the screen basket. A part of this suspension of precipitated silica is recirculated via the line and the pump, in order to further reduce the grit and nodule portion.

[0037] The suspension of precipitated silica has a pH of 3.2. The solid content is 19.0 wt%.

[0038] The suspension of precipitated silica is then spray dried, whereby a one-component nozzle (spray tower) is used.

[0039] The spray drying conditions and the physicochemical data of the precipitated silica granules thus obtained are presented in Table 1.

Table 1

A. Trocknungsbedingungen A)		3	6	7	8	9
Versuchsnummer 1)		3	6	7	8	9
Produktbezeichnung 2)		3)rodukt A	3)rodukt A	3)rodukt A	3)rodukt A	3)odukt A
Düsendruck 4)	[bar]	8	12	12	12	12
Prozeßluftmenge 5)	[kg/h]	654	654	512	509	509
6)Eintrittstemperatur	[°C]	384	384	464	428	399
Austrittstemperatur 7)	[°C]	175	171	159	159	151
8)Dichte Suspension	[g/ml]	1,1	1,1	1,1	1,1	1,1
Suspensionstemperatur 9)	[°C]	20	20	20	20	20
Feststoffgehalt 10)	[%]	19	19	19	19	19
Viskosität 11)	[mPa*s]	75	75	75	75	75
B. Physikalisch-chemische Eigenschaften B)						
Produktfeuchte 12)	[%]	61	23	23	33	43
Korngrößenverteilung 13)						
D[v, 0.5]	[μm]	480	314	380	349	387
D[v, 0.9]	[μm]	907	467	485	476	484
D[v, 0.1]	[μm]	305	209	235	232	265
N₂-Oberfläche 14)	[m²/g]	200	201	199	203	199
15) TAB-Oberfläche	[m²/g]	180	178	179	180	178
pH-Wert 16)		5	4,9	5,1	5,2	5
17)itfähigkeit	[μS]	600	510	630	605	580
Feuchte 18)	[%]	4,1	5,1	4,2	3,8	4,2
Stampfgewicht 19)	[g/l]	278	263	263	270	278
20)BP-Aufnahme	[g/100g]	217	250	247	241	233
Alpine SR > 500 μm 21)	[%]	22,9	0,3	0,6	0,5	0,4
22)lpine SR > 355 μm	[%]	69,7	-	27,7	31,5	30
Alpine SR > 250 μm 23)	[%]	93,8	74	81,2	81,2	84,5
24)pine SR > 180 μm	[%]	98	92	96	93,8	95,8
Alpine SR > 63 μm 25)	[%]	100	99	100	100	100
26)ine SR > 45 μm	[%]	100	100	100	100	100

Key to Table 1:
A) drying conditions
B) physicochemical properties
1) experiment number
2) name of description
3) product A
4) nozzle pressure
5) process air quantity
6) inlet temperature

7)   outlet temperature
8)   suspension density
9)   suspension temperature
10)   solid content
11)   viscosity
12)   product moisture
13)   grain size distribution
14) N_2 surface
15)   CTAB surface
16)   pH
17)   conductivity
18)   moisture content
19)   tapped weight
20)   DBP sorption
21)   Alpine SR > 500 μm
22)   Alpine SR > 355 μm
23)   Alpine SR > 250 μm
24)   Alpine SR > 180 μm
25)   Alpine SR > 63 μm
26)   Alpine SR > 45 μm

Example 2

Preparation of the precipitated silica granules

(product B)

[0040] The precipitation silica is prepared as in DE-A 31 44 299 example 5. The process is as follows:

[0041] 60 m^3 water at a temperature of 40°C is placed in a 75 m^3 wooden vat, which serves as precipitation tank and which is equipped with a MIG crossbar stirrer and an Ekato turbine shearer. Commercially available water glass (SiO_2, 26.8 wt% Na_2O: 8.0 wt%, modulus = 3,35) at a rate of 9.8 m^3/h and concentrated sulfuric acid (96%) at a rate of 0.98 m^3/h also flow into this vat at the same time. In the process, the acid is added via the turbine, which is put into

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operation when the precipitation begins. As it is added, the pH of the precipitation material is kept at 6.0. After the 13th minute of precipitation, i.e. when a rise in viscosity begins to show, the addition of water glass and acid is interrupted for 90 minutes. During this interruption phase, the Ekato turbine continues to shear. Beginning at minute 103, the addition of water glass is continued, maintaining the above-mentioned addition rate and pH, until minute 146. The solid content of the precipitate suspension is then 46 g/l. The temperature can then have reached a value of 42 – 49°C, depending on the external temperature conditions. The final pH is 6.0. Overall, 9.1 m^3 water glass and 0.91 m^3 sulfuric acid enter into reaction. The suspension is aged in an intermediate container before being pressed for 17 hours. Following this aging phase, the suspension is filtered using 4 filter presses. The fill time in this case is 1 hour at a final pressure of 3.3 bar. Following a very brief washing time of just 1.5 hours, a conductivity value of the draining filtrate of 1,050 µS is found, after 4 hours of washing the value is 280 µS.

[0042] The solid content of the filter cake that is obtained is 16.5 – 17 wt%.

[0043] The washed precipitation silica filter cake is transferred into a suspension of precipitated silica, as in example 1 of DE-C 24 47 613. The process is as follows:

[0044] 260 kg/min precipitation silica filter cake with a water content of 82% is fed in by means of the twin screw at 295 rpm

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through the constricted discharge to increase the kneading/mixing effect at the same time as 0.6 liter/min dilute, aqueous 30% sulfuric acid from the line, into the 1.5 m^3 dissolver container. During this process, the acid concentration is kept such that the suspension of precipitated silica has a pH of 3. The dissolver container has a diameter of 1.2 m, a height of 1.5 m, and is cylindrical in shape. The dissolver, which is a double disk dissolver, has a diameter of both disks of 0.4 m and is driven by an electric motor, which has a power of 36.7 kW (50 HP). The rotational speed of the two disks is 21 m/s (1,000 rpm). The submersible probes are used to keep the filling level between 0.8 and 1.20 m, so that 1.5 kg suspension of precipitated silica is in the dissolver container per unit of 1 kg precipitation silica filter cake. The suspension of precipitated silica is removed from the zone of greatest turbulence using the screen basket. A part of this suspension of precipitated silica is recirculated via the line and the pump, in order to further reduce the grit and nodule portion.

[0045] The suspension of precipitated silica has a pH of 2.9. The solid content is 17.5 wt%.

[0046] The suspension of precipitated silica is then spray dried, whereby a one-component nozzle (spray tower) is used.

[0047] The spray drying conditions and the physicochemical data of the precipitated silica granules thus obtained are presented in Table 2.

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Table 2

A. Trocknungsbedingungen A			
Versuchsnummer 1		17	18
2 Produktbezeichnung		3 Produkt B	3 Produkt B
Düsendruck 4	[bar]	14	14
Prozeßluftmenge 5	[kg/h]	512	519
6 Eintrittstemperatur	[°C]	446	407
Austrittstemperatur 7	[°C]	160	151
8 ichte Suspension	[g/ml]	1,1	1,1
Suspensionstemperati 9	[°C]	20	20
Feststoffgeha 10	[%]	17,5	17,5
Viskosität 11	[mPa*s]	75	75
B. Physikalisch-chemische Eigenschaften B			
Produktfeuchte 12	[%]	10	39
Korngrößenverteilung 13			
D[v, 0.5]	[µm]	332	400
D[v, 0.9]	[µm]	457	488
D[v, 0.1]	[µm]	223	261
N_2-Oberfläche 14	[m²/g]	430	423
15 AB-Oberfläche	[m²/g]	340	337
pH-Wert 16		4	3,8
Leitfähigkeit 17	[µS]	680	660
Feuchte 18	[%]	4,5	4,4
Stampfgewicht 19	[g/l]	204	206
20 BP-Aufnahme	[g/100g]	297	282
Alpine SR > 500 µm 21	[%]	0,2	0,5
22 pine SR > 355 µm	[%]	17,6	30,3
Alpine SR > 250 µm 23	[%]	78,4	85
24 pine SR >180 µm	[%]	95,7	97,4
Alpine SR > 63 µm 25	[%]	100	100
26 ine SR > 45 µm	[%]	100	100

Key to Table 2:
A)   drying conditions
B)   physicochemical properties
1)   experiment number
2)   name of description
3)   product B
4)   nozzle pressure
5)   process air quantity
6)   inlet temperature

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7)   outlet temperature
8)   suspension density
9)   suspension temperature
10)   solid content
11)   viscosity
12)   product moisture
13)   grain size distribution
14) N_2-surface
15)   CTAB surface
16)   pH
17)   conductivity
18)   moisture content
19)   tapped weight
20)   DBP sorption
21)   Alpine SR > 500 μm
22)   Alpine SR > 355 μm
23)   Alpine SR > 250 μm
24)   Alpine SR > 180 μm
25)   Alpine SR > 63 μm
26)   Alpine SR > 45 μm

Example 3

Preparation of the precipitated silica granules

(product C)

[0048] The precipitation silica is prepared as in DE-A 195 26 476 example 2. The process is as follows:

[0049] 1,725 kg sodium silicate solution (8.90 wt% Na_2O; 27.73 wt% SiO_2 with modulus $SiO_2:Na_2O = 3.22$), and 56.8 m^3 hot water are mixed in a 75 m^3 container and set at 93°C. The alkali number of the receiver material (use of 1n HCl per 100 ml solution against phenolphthalein) is 7.0.

[0050] During the following 90 minutes, with stirring and while maintaining a constant alkali number of 7, 16,076 kg sodium silicate

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solution (8.90 wt% Na_2O and 27.73 wt% SiO_2; modulus $SiO_2:Na_2O$ = 3.22; temperature 57°C) and 2,168 kg 94% sulfuric acid at 91 – 93°C are added at the same time to the receiver. The addition of sodium silicate solution is then stopped. The addition of 94% sulfuric acid is continued for 30 minutes with continued stirring until the pH of the precipitate suspension has been set at 3.0. The solid content of the suspension of precipitated silica thus obtained is 72 g/l. The suspension is diluted with 38,000 liters of water and the precipitation silica is separated using a filter press and washed out with water.

[0051] The washed precipitation silica filter cake is transferred into a suspension of precipitated silica, as in example 1 of DE-C 24 47 613. The process is as follows:

[0052] 260 kg/min precipitation silica filter cake with a water content of 82% is fed in by means of the twin screw at 295 rpm through the constricted discharge to increase the kneading/mixing effect at the same time as 0.6 liter/min dilute, aqueous 30% sulfuric acid from the line, into the 1.5 m^3 dissolver container. During this process, the acid concentration is kept such that the suspension of precipitated silica has a pH of 3. The dissolver container has a diameter of 1.2 m, a height of 1.5 m, and is cylindrical in shape. The dissolver, which is a double disk dissolver, has a diameter of both disks of 0.4 m and is driven by an electric motor, which has a power of 36.7 kW (50 HP). The rotational speed of the two disks is

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21 m/s (1,000 rpm). The submersible probes are used to keep the filling level between 0.8 and 1.20 m, so that 1.5 kg suspension of precipitated silica is in the dissolver container per unit of 1 kg precipitation silica filter cake. The suspension of precipitated silica is removed from the zone of greatest turbulence using the screen basket. A part of this suspension of precipitated silica is recirculated via the line and the pump, in order to further reduce the grit and nodule portion.

[0053] The suspension of precipitated silica has a pH of 2.8. The solid content is 19 wt%.

[0054] The suspension of precipitated silica is then spray dried, whereby a one-component nozzle (spray tower) is used.

[0055] The spray drying conditions and the physicochemical data of the precipitated silica granules thus obtained are presented in Table 3.

Table 3

A. Trocknungsbedingungen [A]				
Versuchsnummer [1]		16	13	14
Produktbezeichnung [2]		[3]Produkt C	[3]Produkt C	[3]Produkt C
Düsendruck [4]	[bar]	13	15	16
Prozeßluftmenge [5]	[kg/h]	509	522	509
Eintrittstemperatur [6]	[°C]	384	428	391
Austrittstemperat. [7]	[°C]	161	170	159
[8]chte Suspension	[g/ml]	1,1	1,1	1,1
Suspensionstemperat [9]	[°C]	20	20	20
Feststoffgehalt [10]	[%]	19	19	19
Viskosität [11]	[mPa*s]	74	74	74
B. Physikalisch-chemische Eigenschaften [B]				
Produktfeuchte [12]	[%]	37	46	29
Korngrößenverteilung [13]				
D[v, 0.5]	[µm]	308	-	321
D[v, 0.9]	[µm]	450	-	462
D[v, 0.1]	[µm]	203	-	209
N₂-Oberfläche [14]	[m²/g]	190	188	186
[15]TAB-Oberfläche	[m²/g]	168	170	171
pH-Wert [16]		4	4,1	4
[17]itfähigkeit	[µS]	350	320	350
Feuchte [18]	[%]	3,8	3,8	3,9
Stampfgewicht [19]	[g/l]	276	294	278
[20]BP-Aufnahme	[g/100g]	222	216	231
Alpine SR > 500 µm [21]	[%]	0,1	0,7	0,1
[22]pine SR > 355 µm	[%]	6,8	16,1	6,8
Alpine SR > 250 µm [23]	[%]	64,5	68,1	56,4
[24]pine SR > 180 µm	[%]	86,3	88,8	66
Alpine SR > 63 µm [25]	[%]	100	100	99,1
[26]pine SR > 45 µm	[%]	100	100	100

Key to Table 3:
A) drying conditions
B) physicochemical properties
1) experiment number
2) name of description
3) product C
4) nozzle pressure
5) process air quantity
6) inlet temperature

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7)   outlet temperature
8)   suspension density
9)   suspension temperature
10) solid content
11) viscosity
12) product moisture
13) grain size distribution
14) N_2 surface
15) CTAB surface
16) pH
17) conductivity
18) moisture content
19) tapped weight
20) DBP sorption
21) Alpine SR > 500 μm
22) Alpine SR > 355 μm
23) Alpine SR > 250 μm
24) Alpine SR > 180 μm
25) Alpine SR > 63 μm
26) Alpine SR > 45 μm

Example 4

(FSD Method)

[0056] The precipitation silica is prepared as in DE-A 14 67 019 example 1. The process, in detail, is as in example 1 of the present application.

[0057] The precipitation silica that is obtained is filtered off. The precipitation silica filter cake is washed and transferred into a suspension of precipitated silica, as in example 1 of DE-C 24 47 613. The process, in detail, is as in example 1 of the present application.

[0058] The suspension of precipitated silica has a solid content of 19.2 wt% and a pH of 3.2.

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[0059] Three drying experiments are carried out in the FSD unit with the suspension of precipitated silica. In the process, the suspension of precipitated silica is atomized in the various experiments using three different nozzle sizes. Along with the nozzle geometry that is used, this operating parameter has the greatest influence on the particle size distribution.

[0060] Before drying begins, the suspension that is used is filtered with an oscillating screen having a mesh size of 0.5 mm, in order to prevent clogging of the pressure nozzle by any impurities that may be present when the experiment is carried out. The operating parameters are presented in Table 5:

Table 5

Versuchsbezeichnung 1			FSD 1	FSD 2	FSD 3
Druckdüse Typ 2			SDX 1,6 SB	SDX 1,4 SB	SDX 1,5 SB
Düsendurchmesser 3		mm	1,6	1,4	1,5
Zerstäubungsdruck 4		bar	16,2 - 17,2	16,2 - 17,2	13,3
Suspensionsmenge 5		l/h	156	156	153
Eintrittstemperatur 6		°C	301	300	295
Austrittstemperatur 7		°C	104	108	108
Eintrittstemperatur Fließbett (integriert) 8		°C	125	125	158
Differenzdruck Fließbett (integriert) 9		mmWS	38,5	37,1	43,7
Unterdruck Trockenkammer 10		mmWS	-20	-15	-22
...ferenzdruck Entstaubungsfilter 11		mmWS	147	198	83

Key to Table 5:
1) designation of experiment
2) pressure nozzle type
3) nozzle diameter
4) atomization pressure
5) amount of suspension
6) inlet temperature
7) outlet temperature
8) inlet temperature fluidized bed (integrated)
9) differential pressure, fluidized bed (integrated)
10) vacuum, drying chamber

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11)   differential pressure dust filter

[0061] The chemical-physical data are then determined from the respective average samples of the precipitation silica granules that have been obtained. They are presented in Table 6:

Table 6

		Produkt A Versuch 1 FSD-Anlage [1]	Produkt A Versuch 3 FSD-Anlage [2]
Wassergehalt [3]	%	6,3	4,7
pH-Wert [4]		4,0	3,9
Leitfähigkeit [5]	µS	930	700
[6] N_2-Oberfläche	m^2/g	-	207
CTAB-Oberfläche [7]	m^2/g	-	183
[8] DBP-Aufnahme	g/100 g	242	239
Stampfdichte [9]	g/l	270	278
[10] pine-SR > 63 µm	%	99	100
Alpine-SR > 180 µm [11]	%	98	98
[12] pine-SR > 250 µm	%	89	89
Alpine-SR > 300 µm [13]	%	52	63
[14] pine-SR > 500 µm	%	6,0 *)	1,6
Trübung [15]		-	1,0

*) This fraction contains caking on the walls from the drying
    chamber.

Key to Table 6:
1)   product A test 1 FSD unit
2)   product A test 3 FSD unit
3)   water content
4)   pH
5)   conductivity
6) N_2 surface
7)   CTAB surface
8)   DBP sorption
9)   tapped bulk density
10) Alpine-SR > 63 µm
11) Alpine-SR > 180 µm
12) Alpine-SR > 250 µm
13) Alpine-SR > 300 µm
14) Alpine-SR > 500µm

15) turbidity

[0062] The grain distributions determined for the precipitated silica granules produced using the FSD unit are shown graphically in Figure 2, as compared to a typical Sipernat 22 sample. The coarseness and lack of dust in the precipitated silica granules examined are clearly seen over the course of the curves.

[0063] The FSD method is described in Chem. Ing. Techn. 59 (1987) No. 2, pp. 112 - 117.

[0064] FSD means Fluidized Spray Dryer and describes a spray dryer with an integrated fluidized bed dryer.

[0065] Dust measurements are made with the precipitated silica granules A, B, and C, as well as FSD.

[0066] The numbers of the precipitated silica granules A, B, C, and FSD itself and of the products A, B, C, and FSD charged with vitamin E acetate and with choline chloride solution are determined.

[0067] The results of the dust measurements are presented in Tables 7, 8, and 9.

[0068] The dust measurements on the precipitated silica granules are carried out by the following method:

Basics

[0069] Processing and handling precipitation silica produces dust in various forms (total dust, fine dust, ultra-fine dust). These dusts differ, depending on the fine and ultra-fine percentages of the

precipitation silica. Measurement of the dust curve shows the maximum

dust and the settling behavior of the precipitation silica.

Testing means

A. Test devices:

[0070]

dust meter "Cassella," manufactured by Hoechst
recorder, manufactured by ABB
precision balance
screw attachment with funnel and slide
250 ml glass powder bottle

Implementation

[0071] The precipitation silicas are classified in the following

categories, according to their particle size:

[1] Fällungskieselsäuren		[2] Teilchengröße, Mittlere [μm]	[3] Bestimmung des Staubverhaltens
A	sehr kleine Teilchengröße [4]	ca. 5,5	von 30 g Einwaage wird die Staubzahl bestimmt und von 15 - 20 g die vollständige Staubkurve aufgenommen [5]
		ca. 4,5	
		ca. 5,5	
B	kleine Teilchengröße [6]	ca. 7	von 30 g Einwaage wird die Staubzahl bestimmt und von 20 g die vollständige Staubkurve aufgenommen [7]
		ca. 8	
		ca. 15	
C	mittlere Teilchengröße [8]	ca. 100	von 30 g Einwaage wird die vollständige Staubkurve aufgenommen [9]
		ca. 185	
		ca. 50	
D	grobe Teilchengröße [10]	ca. 200	von 30 g Einwaage wird die Staubzahl bestimmt und von 20 g bzw. vorzugsweise 45 g die vollständige Staubkurve aufgenommen [11]

Key to Table (p. 12):
1)   Precipitation silicas
2)   Particle size, average [μm]
3)   Determination of dust behavior
4)   very small particle size
5)   the dust number is determined for a 30 g weighed portion and the
     complete dust curve is taken for 15 – 20 g
6)   small particle size

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7)   the dust number is determined for a 30 g weighed portion and the
     complete dust curve is taken for 20 g
8)   average particle size
9)   The entire dust curve is taken for 30 g weighed portion
10) coarse particle size
11) the dust number is determined for a weighed portion of 30 g and
     the complete dust curve is taken for 20 g or, preferably, 45 g

[0072] The dust number of the precipitation silicas A, B, and D

at 30 g is determined as follows:

[0073] 30 g of the precipitation silica under examination is

weighed into a 250 ml screw-top jar and quantitatively transferred

into the device's funnel by means of a funnel (screw attachment).

After the device is nulled, the recorder is started at a rate of

6 cm/min and the measurement begun with the start button on the dust

meter.

[0074] The sample is automatically transferred into the downpipe

by means of a magnetic switch. The maximum value is displayed

digitally after 3 seconds. After 30 seconds, a second value is

determined, which is added to the maximum value. The sum is the dust

number, which is also displayed digitally on the device.

[0075] Determination of the dust curve and dust number:

[0076] The amount of precipitation silica under test indicated

in the table is weighed into a 250 ml screw-top jar and the dust

number determined as above. In addition, the settling behavior is

observed over a measurement period of 5 minutes. The test is then

ended and the recorder curve assessed.

Evaluation

[0077] For the precipitation silicas in category C, the measured results include the measured values, in %, after 10 seconds, 30 seconds, 1 minute, and 5 minutes, the maximum and the dust number, and the settling times of the dust at values of 5% and 2%, indicated in seconds.

[0078] The precipitation silicas of categories A, B, and D, with which the measurements were carried out with 15, 20, and 45 g, respectively, the amounts must be converted to the comparison amount of 30 g. For this purpose, the dust number of the sample with a weighed portion of 30 g is divided by the dust number for the weighed portion x g. The factor determined in this way is then used for the subsequent conversions and, in this way, the dust behavior of a 30 g weighed portion is calculated.

calculated value at 30 g = value at xg * dust number 30 g / dust number xg

[0079] When indicating results, a footnote should always indicate whether they are calculated or actually determined values.

Table 7

Fällungskieselsäure [2]	Produkt A Versuch 6, 829 [3]	Produkt B Versuch 18, 830 [4]	Produkt C Versuch 13, 831 [5]	Produkt A, FSD Versuch 3, 832 [6]
Staubmessungen Fällungskieselsäuregranulate ohne Belastung [1]				
Maximum, % [7]	6,6 **	3,5 **	3,2 **	1,2 **
Staubzahl % [8]	10,1 **	5,5 **	4,1 **	2,0 **
Wert bei, % [9]				
10 Sek. [10]	4,6 **	2,7 **	1,8 **	1,0 **
30 Sek. [11]	3,5 **	2,0 **	0,9 **	0,8 **
1 Min. [12]	2,9 **	0,9 **	0,8 **	0,6 **
5 Min. [13]	0,7 **	0,6 **	0,4 **	0,1 **
Absinkzeit bis 5%, Sek. [14]	3	2	2	0
Absinkzeit bis 2 %, Sek. [15]	29	32	16	0

(** 45 g measurement at 30 g calculated)

Key to Table 7:
1)   dust measurements precipitated silica granules without load
2)   precipitation silica
3)   product A test
4)   product B test
5)   product C test
6)   product A, FSD test
7)   maximum, %
8)   dust number %
9)   value at, %
10)   10 s
11)   30 s
12)   1 min
13)   5 min
14)   settling time to 5%, s
15)   settling time to 2%, s

[0080] The precipitated silica granules of this invention (48 g)

are loaded with 90% vitamin E acetate (52 g).

[0081] The dust behavior is determined by the same method as

with the precipitated silica granules. The results are presented in

Table 8:

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Table 8

Staubmessungen nach der Beladung mit Vitamin E-Acetat ohne Belastung				
Fällungskieselsäure	Produkt A Versuch 6, 829	Produkt B Versuch 18, 830	Produkt C Versuch 13, 831	Produkt A, FSD Versuch 3, 832
Maximum, %	2,7	4,2	1,6	1,2
Staubzahl %	3,9	5,9	2,1	1,4
Wert bei, %				
10 Sek.	2,1	2,8	0,8	0,6
30 Sek.	1,2	1,7	0,5	0,2
1 Min.	0,8	1,2	0,3	0,2
5 Min.	0,2	0,3	0,1	0
Absinkzeit bis 5%, Sek.	0	0	0	0
Absinkzeit bis 2%, Sek.	12	24	2	0

Key to Table 8:
1) dust measurements after loading with vitamin E acetate without load
2) precipitation silica
3) product A test
4) product B test
5) product C test
6) product A, FSD test
7) maximum, %
8) dust number %
9) value at, %
10) 10 s
11) 30 s
12) 1 min
13) 5 min
14) settling time to 5%, s
15) settling time to 2%, s

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Table 9

50 %iges Cholinchlorid [1]				
Fällungskieselsäure [2]	Produkt A Versuch 6, 829 [3]	Produkt B Versuch 18, 830 [4]	Produkt C Versuch 13, 831 [5]	Produkt A, FSD Versuch 3, 832 [6]
Stampfdichte, g/l [7]	677	590	634	714
Schüttkegelhöhe, mm [8]	23	11	42	12
Glasauslaufgefäße, Note [9]	2	1	5	1
Zugabe Sipernat D17, % [10]	0,1	/	0,1	/
Schüttkegelhöhe, mm [11]	9	/	11	/
Glasauslaufgefäße, Note [12]	1	/	1	/
Agglomeratanteil, % [13]	1,1	0,6	zu feucht	1,9
maximale Cholinchloridaufnahme, g/100g [14]	245 *	300 *	205 *	240 *

*Due to the particle size that is produced, the choline chloride
concentrates are very flowable, despite their waxy appearance.
For this reason, the values of the maximum choline chloride
sorption are very high.

Key to Table 9:
1)   50% choline chloride
2)   precipitation silica
3)   product A test
4)   product B test
5)   product C test
6)   product A, FSD test
7)   tapped bulk density, g/l
8)   pouring-cone height, mm
9)   glass outlet receptacles
10)   addition of Sipernat D17, %
11)   pouring-cone height, mm
12)   glass outlet receptacles, grade
13)   agglomerate portion, %
14)   too moist
15)   maximum choline chloride sorption, g/100g

[0082] The precipitated silica granules of this invention are

loaded with 75% aqueous choline chloride solution. The data for the

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loaded precipitated silica granules are presented in Table 9. The following methods are used to determine the data:

Determination of tapped bulk density

Basics

[0083] One parameter for the filling of silos is the tapped bulk density. It provides information on the maximum mass of a material that can be filled into the silo per available unit of volume.

Testing means

A. Test devices

[0084]

graduated cylinder, 250 ml, manufactured by Engelsmann, Ludwigshafen

tap volume meter, manufactured by Engelsmann, Ludwigshafen

funnel

sheet

precision balance

Implementation

[0085] A filling level of 200 ml ± 10 ml of unsifted material is placed in a 250 ml graduated cylinder and then tapped 1,250 times in the tap volume meter. After tapping, the surface in the graduated cylinder is smoothed out with a sheet and the volume read.

Evaluation

[0086]

tapped bulk density in [g/l] = [mass [g] x 1,000]/ [volume [ml]]

Testing flowability using pouring-cone height

Basics

[0087] Good flow properties on the part of the powder in question are required for smooth emptying of a powder silo and for precise metering of the powder. The angle of repose is a good criterion for assessing the flow properties. Since the pouring-cone height with the same base measurement is directly dependent on the angle of repose and is much easier to determine, it is determined. Powders with good flowability have a low pouring-cone height.

Testing means

A. Test devices

[0088]

metal sieve (1 mm)

marking gauge

metal solid cylinder, d = 50 mm, h = 80 mm

scraper

Implementation

[0089] The wire sieve is attached to the stand about 10 cm over the solid metal cylinder. In order to set the final height of the sieve, the powder that is assumed to be the worst is slowly poured onto the sieve and carefully passed through the sieve using a scraper. The distance from the sieve to the point of the pouring cone of powder is set to 2 cm. This height applies to all subsequent comparison tests. If the pouring cone of powder is uniformly shaped,

then the screening of the powder is over and the pouring-cone height is read with the marking gauge at the apex of the cone.

<u>Evaluation</u>

[0090] The pouring-cone height is indicated in mm.

<u>Testing the flowability using glass outlet receptacles</u>

<u>Basics</u>

[0091] Good flow properties (of the powder in question) are required for smooth emptying of a powder silo and for precise metering of the powder. Powders with poor flowability do not flow or flow only through outlet receptacles with very large outlet openings; for this reason, powders with good flowability are given a low grading.

<u>Testing means</u>

<u>A. Test devices</u>

[0092]

measuring vessel 1 = 2.5 mm diameter

measuring vessel 2 = 5.0 mm diameter

measuring vessel 3 = 8.0 mm diameter

measuring vessel 4 = 12.0 mm diameter

measuring vessel 5 = 18.0 mm diameter

<u>Implementation</u>

[0094] Starting with measuring vessel 5, the outlet receptacle is placed on a sheet of weighing paper and, using a second weighing paper serving as a funnel, it is filled to its top edge with the

powder that is to be tested. With careful lifting, the opening is uncovered in such a way that the powder can flow out. If the powder flows through one vessel, then the next smaller vessel is used and the procedure indicated above is repeated.

Evaluation

[0094]

Grade	Assessment
1	powder flows freely out of vessel no. 1
2	powder flows freely out of vessel no. 2
3	powder flows freely out of vessel no. 3
4	powder flows freely out of vessel no. 4
5	powder flows freely out of vessel no. 5
6	powder does not flow out of vessel no. 5

<u>Test of the agglomerate portion of a 50% choline chloride concentrate</u>

<u>Basics</u>

[0095] When spray-dried precipitation silica is mixed with 75% choline chloride solution for producing a 50% concentrate, various workability values of the precipitation silicas can be determined. In practice, a non-homogenizable agglomerate portion is troublesome. This is caused, in particular, by insufficient sorption characteristics and high fine and ultra-fine portions in the precipitation silica.

<u>Testing means</u>

A. Test devices

[0096]

food processor, manufactured by Braun, model KM 321

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dropping funnel, 250 ml

sieve, 0.5 mm

precision balance

timer

B. Test substances

[0097]

75% choline chloride solution

Sipernat D17 made by Standmuster

precipitation silica to be tested

Implementation

[0099] 200 g of the precipitation silica to be tested is placed in the mixing bowl. While stirring at setting no. 2, 400 g 75% choline chloride solution is added dropwise over a period of 2 minutes. After the drops are added, the material is stirred for 2 minutes. 6 g Sipernat D17 is then added for better separation and stirred for an additional 0.5 minutes. The mixture thus obtained is placed on a 0.5 mm sieve and the powder separated from the agglomerates by shaking. The oversize agglomerate material is weighed out.

Evaluation

agglomerate portion [%] = [residue [g] x 100 [%]] / 600 [g]

Test of the improved flow of a 50% choline chloride concentrate

Basics

[0100] A 50% choline chloride concentrate on precipitation silica is to have the best possible flowability with an addition of as little Sipernat D17 as possible. This is achieved if, by the further addition of Sipernat D17, no improvement is made in the grade of the glass outlet receptacles and the pouring-cone height.

Testing means

A. Test devices

[0101]

analytical balance

precision balance

Turbula mixer

B. Test substances

[0102] 50% choline chloride mixture on precipitation silica

Sipernat D17 under test

Implementation

[0103] 0.1 g Sipernat D17 is added to the 50%n choline chloride concentrate and mixed for 2 minutes in the Turbula mixer at medium speed. The flowability of this mixture is determined using its pouring-cone height and glass outlet receptacles. 0.1 g Sipernat D 17 amounts are added until a flow grading of 1 is achieved. If the grading 1 is not reached, an additional 0.1 g Sipernat D17 is added and the tests are repeated.

<u>Evaluation</u>

[0104] Pouring-cone height in mm at x g of added Sipernat D17. Glass outlet receptacles in flow grading at x g of added Sipernat D17.

<u>Testing the maximum choline chloride sorption</u>

<u>Basics</u>

[0105] Determination of the maximum choline chloride sorption should include the total sorption potential of the precipitation silica.

<u>Testing means</u>

A. Test devices

[0106]

250 ml glass beaker, tall

spatula

precision balance

B. Test substances

75% choline chloride solution

precipitation silica to be tested

<u>Implementation</u>

[0107] 10 g of the precipitation silica to be tested is placed in a tall 250 ml glass beaker and 75% choline chloride solution is added dropwise during stirring with the spatula. While constantly observing the mixture, the time of maximum sorption is determined. If closely observed, white precipitation silica particles can be

observed, which clearly lift off of waxy (saturated) particles. The maximum choline chloride sorption is achieved when no more unloaded particles are found in the mixture and the mixture is not yet waxy/lubricating.

Evaluation

[0109]

Max. choline chloride sorption in g/100 g = [(a−10) x 100]/ 10

a = total weight

[0110] This performance-based examination shows that the precipitated silica granules of this invention have excellent grain stability and, thus, a very low tendency to form dust.

[0111] Particularly low in dust and, thus, having stable grains are precipitated silica granules that are prepared by the FSD method.

[0112] The precipitated silica granules of this invention are vary well suitable for use as carriers for choline chloride and vitamin E.

[0113] For comparison purposes, a known precipitated silica granule in accordance with EP-B 0 018 866 (Zeosil 1165 MP) is studied by the same methods. The results are presented in Tables 10, 11, and 12.

Table 10

	Zeosil 1165 MP UB 14624, Muster 858 Rhone Poulenc [1]	Zeosil 1165 MP UB 14502, Muster 775 Rhone Poulenc [2]
Rohware [3]		
Stampfdichte, g/l [4]	259	307
Schüttkegelhöhe, mm [5]	15	16
Glasauslaufgefäße, Note [6]	1	1
Rohware ohne Belastung, 45 g Messung auf 30 g berechnet [7]		
Maximum, % [8]	23,3	26,8
Staubzahl, % [9]	32,4	37,2
Wert bei, % [10]		
10 Sek. [11]	14,0	17,1
30 Sek. [12]	9,1	10,4
1 Min. [13]	5,6	8,1
5 Min. [14]	1,6	2,2
Absinkzeit bis 5 %, Sek. [15]	62	173
Absinkzeit bis 2 %, Sek. [16]	260	> 300

Key to Table 10:
1)   Zeosil 1165 MP UB 14624, Sample 858 Rhone Poulenc
2)   Zeosil 1165 MP UB 14502, Sample 775 Rhone Poulenc
3)   raw material
4)   tapped bulk density, g/l
5)   pouring-cone height, mm
6)   glass outlet receptacles, grade
7)   raw material without load, 45 g measurement at 30 g calculated
8)   maximum, %
9)   dust number, %
10) value at, %
11) 10 s
12) 30 s
13) 1 min
14) 5 min
15) settling time to 5%, s
16) settling time to 2%, s

Table 11

	Zeosil 1165 MP UB 14624, Muster 858 Rhone Poulenc [1]	Zeosil 1165 MP UB 14502, Muster 775 Rhone Poulenc [2]
50 %iges Vitamin E-Konzentrat [3]		
Sauggeschwindigkeit [4]	3	3
Staubverhalten [5]	2	2
Stampfdichte, g/l [6]	577	559
Schüttkegelhöhe, mm [7]	13	12
Glasauslaufgefäße, Note [8]	1	2
50 %iges Vitamin E-Konzentrat ohne Belastung [9]		
Maximum, % [10]	4,3	3,4
Staubzahl, % [11]	5,9	4,7
Wert bei, % [12]		
10 Sek. [13]	2,3	1,6
30 Sek. [14]	1,6	1,3
1 Min. [15]	0,9	0,6
5 Min. [16]	0,2	0,3
Absinkzeit bis 5 %, Sek. [17]	0	0
Absinkzeit bis 2%, Sek. [18]	17	7

Key to Table 11:
1)   Zeosil 1165 MP UB 14624, Sample 858 Rhone Poulenc
2)   Zeosil 1165 MP UB 14502, Sample 775 Rhone Poulenc
3)   50% vitamin E concentrate
4)   sorption rate
5)   dust behavior
6)   tapped bulk density, g/l
7)   pouring-cone height
8)   glass outlet receptacles
9)   50% vitamin E concentrate without load
10)   maximum, %
11)   dust number, %
12)   value at, %
13)   10 s
14)   30 s
15)   1 min
16)   5 min
17)   settling time to 5%, s
18)   settling time to 2%, s

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Table 12

	Zeosil 1165 MP UB 14624, Muster 858 Rhone Poulenc [1]	Zeosil 1165 MP UB [2] 14502, Muster 775 Rhone Poulenc
50 %iges Cholinchlorid [3]		
Stampfdichte, g/l [4]	[5] zu feucht	[5] zu feucht
Schüttkegelhöhe, mm [6]	> 50	> 50
[7] Glasauslaufgefäße, Note	6	6
Zugabe Sipernat D17, % [8]	0,1/0,2/0,3/0,4/0,5/0,6	0,1/0,2/0,4/0,5
[9] Schüttkegelhöhe, mm	>50/>50/>50/14*/13*/13*	>50/>50/22/11
Glasauslaufgefäße, Note [10]	6/6/6/2*/2*/1*	6/6/4**/1
[11] maximale Cholinchloridaufnahme, g/100 g	195	180
Agglomeratanteil, % [12]	zu feucht [5]	zu feucht [5]

Key to Table 12:
1)   Zeosil 1165 MP UB 14624, Sample 858 Rhone Poulenc
2)   Zeosil 1165 MP UB 14502, Sample 775 Rhone Poulenc
3)   50% choline chloride
4)   tapped bulk density, g/l
5)   too moist
6)   pouring-cone height, mm
7)   glass outlet receptacles, grade
8)   addition of Sipernat D17, %
9)   pouring-cone height, mm
10) glass outlet receptacles, grade
11) maximum choline chloride sorption, g/100 g
12) agglomerate portion,%

[0114] The known precipitated silica granule Zeosil 1165 MP in

accordance with EP-B 0 018 866 has the physicochemical

characteristics presented in Table 13.

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Table 13

		Zeosil 1165 MP Rhône Poulenc UB14624 [1]	Zeosil 1165 MP Rhône Poulenc UB14502 11/95 [2]
Spezifische Oberfläche (Ströhlein) [3]	m²/g	157	156
Stampfdichte (DIN ISO 787/11) [4]	g/l	299	307
Trocknungsverlust, 2 h/105 °C (DIN ISO 787/11) [5]	%	6,6	6,9
Glühverlust (ISO 3262/11) [6]	%	4,3	4,0
pH-Wert, 5 % (DIN ISO 787/9) [7]		6,8	7,6
Leitfähigkeit, 20 °C [8]	µS/cm	665	660
DBP-Aufnahme (Originalmaterial) [9]	g/100 g	199	198
DBP-Aufnahme (wasserfrei Korrekturtabelle) [10]	g/100 g	234	234
Teilchengröße, Cilas HR 850, d50%-Wert 0'US [11]	µm	228	207
Teilchengröße, Cilas HR 850, d 5%-Wert 0'US [12]	µm	474	452
Teilchengröße, Cilas HR 850, d95%-Wert 0'US [13]	µm	20	14
Trübung [14]	NTU	8	9
Siebrückstand, Alpine (E=10 g) > 45 µm [15]	%	96	93
Siebrückstand, Alpine (E=10 g) > 63 µm [16]	%	93	91
Siebrückstand, Alpine (E=10 g) > 90 µm [17]	%	92	90
Siebrückstand, Alpine (E=10 g) > 125 µm [18]	%	85	81
Siebrückstand, Alpine (E=10 g) > 150 µm [19]	%	81	78
Siebrückstand, Alpine (E=10 g) > 180 µm [20]	%	69	68
Siebrückstand, Alpine (E=10 g) > 250 µm [21]	%	47	47
Siebrückstand, Alpine (E=10 g) > 315 µm [22]	%	23	24
Siebrückstand, Alpine (E=10 g) > 500 µm [23]	%	0,4	0,5
Siebrückstand, Mocker (E=10 g) > 63 µm [24]	%	< 0,01	0,1
Chlorid, gesamt [25]	ppm	148 ± 1	165

Key to Table 13:
1)   Zeosil 1165 MP Rhône Poulenc UB14624
2)   Zeosil 1165 MP Rhône Poulenc UB14502 11/95
3)   specific surface are (Ströhlein)
4)   tapped bulk density (DIN ISO 787/11)
5)   loss on drying, 2 h/105°C (DIN ISO 787/11)
6)   ignition loss (ISO 3262/11)
7)   pH, 5% (DIN ISO 787/9)
8)   conductivity, 20°C
9)   DBP sorption (original material)
10)   DBP sorption (water-free correction table)
11)   particle size, Cilas HR 850, d50% value 0'US
12)   particle size, Cilas HR 850, d 5% value 0'US
13)   particle size, Cilas HR 850, d95% value 0'US
14)   turbidity

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15)    oversize material, Alpine (E=10 g) > 45µm
16)    oversize material, Alpine (E=10 g) > 63µm
17)    oversize material, Alpine (E=10 g) > 90µm
18)    oversize material, Alpine (E=10 g) > 125µm
19)    oversize material, Alpine (E=10 g) > 150µm
20)    oversize material, Alpine (E=10 g) > 180µm
21)    oversize material, Alpine (E=10 g) > 250µm
22)    oversize material, Alpine (E=10 g) > 315µm
23)    oversize material, Alpine (E=10 g) > 500µm
24)    oversize material, Mocker (E=10 g) > 63µm
25)    chlorides, gesamt
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[0115] The studies show that the known precipitated silica granules cannot be charged with choline chloride. No flowable product is obtained. Moreover, the dust content of the known precipitated silica granule is a multiple higher than that of the precipitated silica granule of this invention.

Claims

1. Precipitated silica granules, characterized by the following dusting parameters:

maximum dust ≤ 5.0 wt%

dust number ≤ 10.0 wt%

settling number to 5% ≤ 5 s

settling number to 2% ≤ 50 s

2. A method for preparing the precipitated silica granules as recited in Claim 1, characterized in that a suspension of precipitated silica is adjusted to a pH of 2 to 3.9 and spray dried.

3. A method for preparing the precipitated silica granules as recited in Claim 2, characterized in that the spray drying is accomplished using a one-component nozzle in a spray tower.

4. A method for preparing the precipitated silica granules as recited in Claim 2, characterized in that the spray drying is accomplished using the FSD method.

5. Use of the precipitated silica granules as recited in Claim 1 for adsorption of liquid agents, in particular vitamins.

6. Use of the precipitation silica as recited in Claim 3 for the adsorption of choline chloride solution.
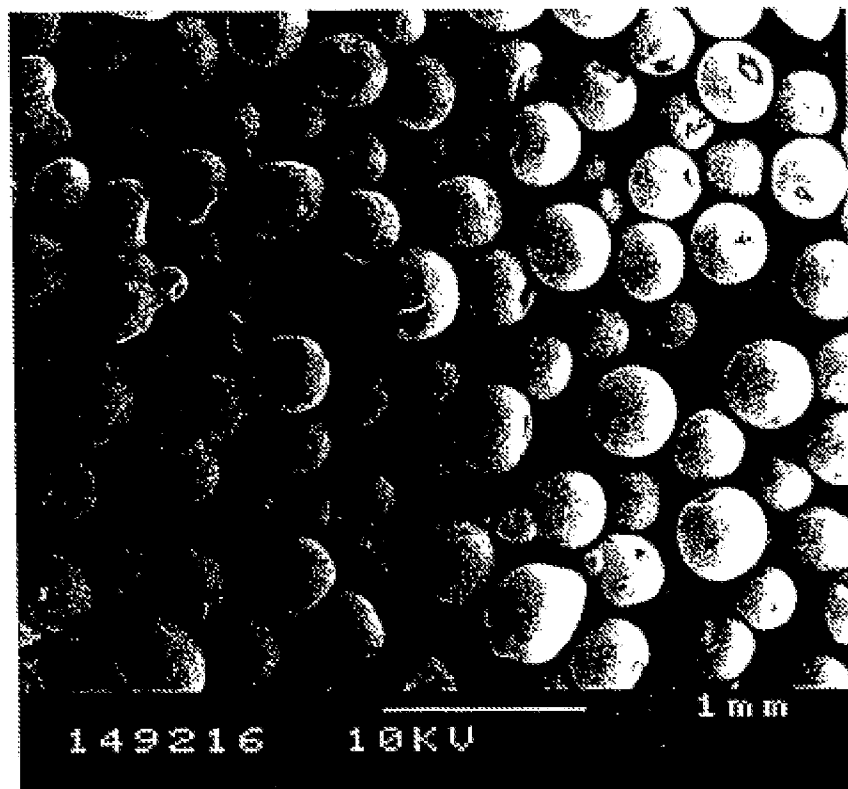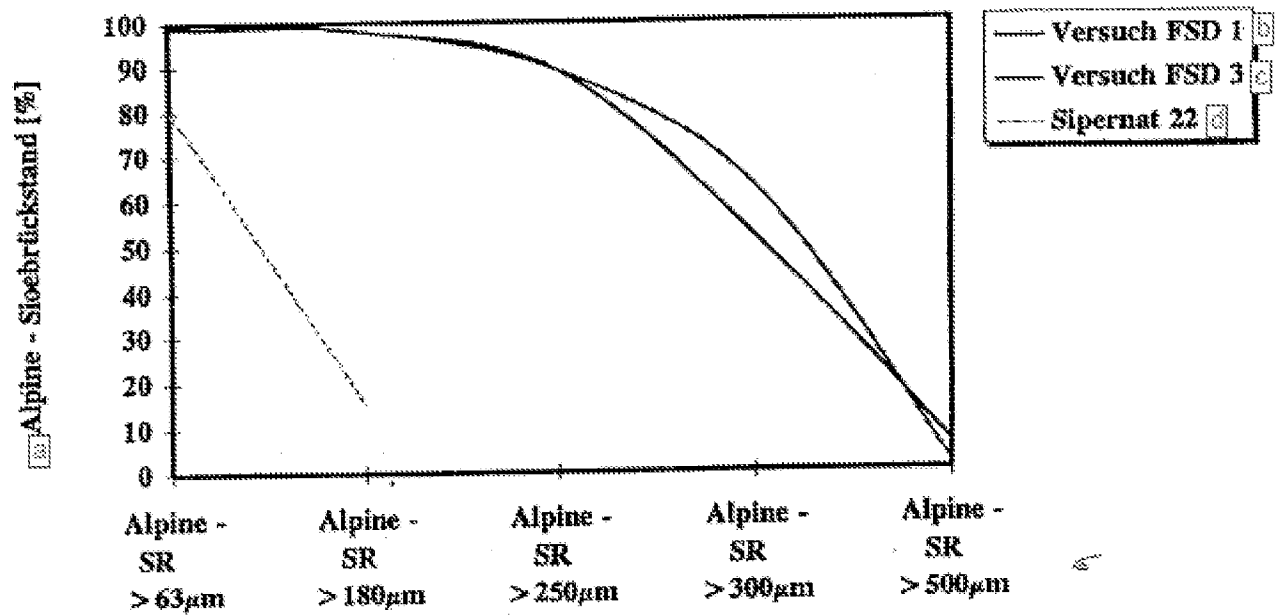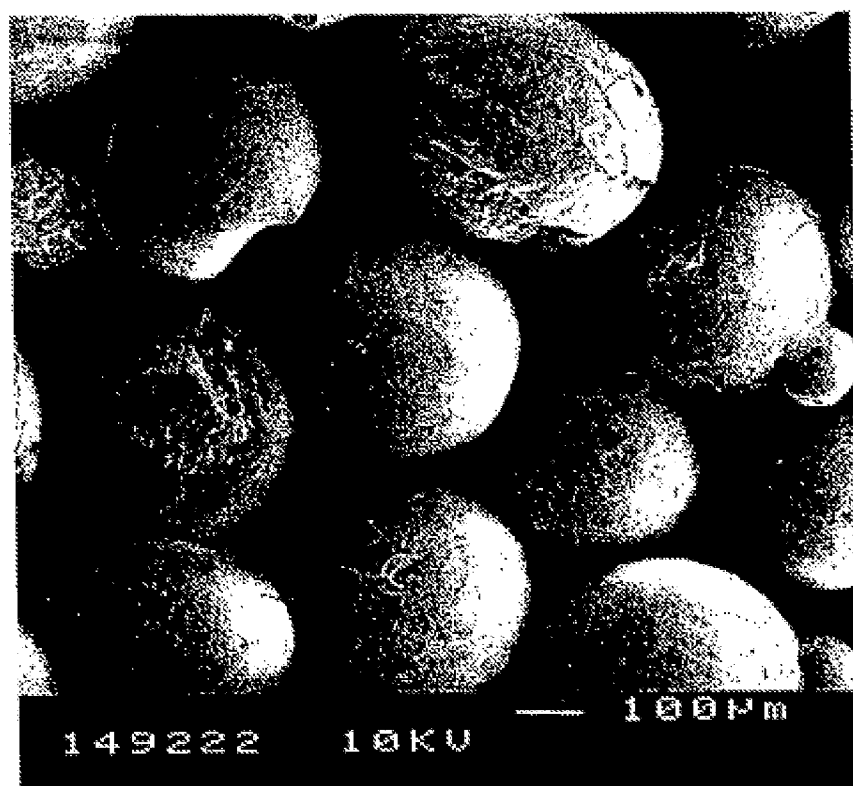
Figur 1

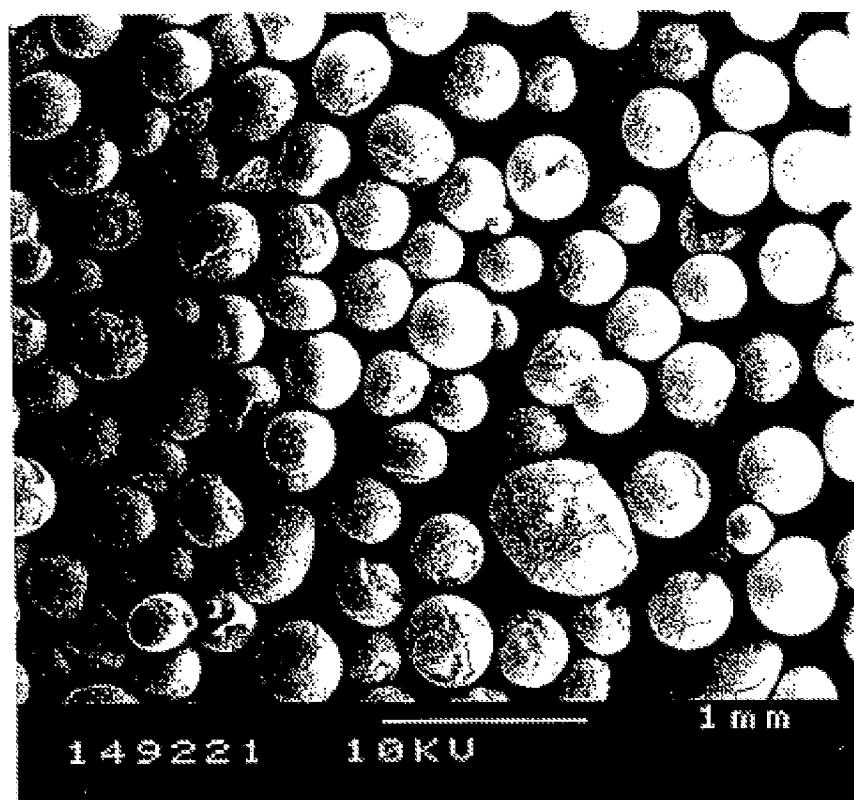149216   10KU   1 mm

149217   10KU   100μm
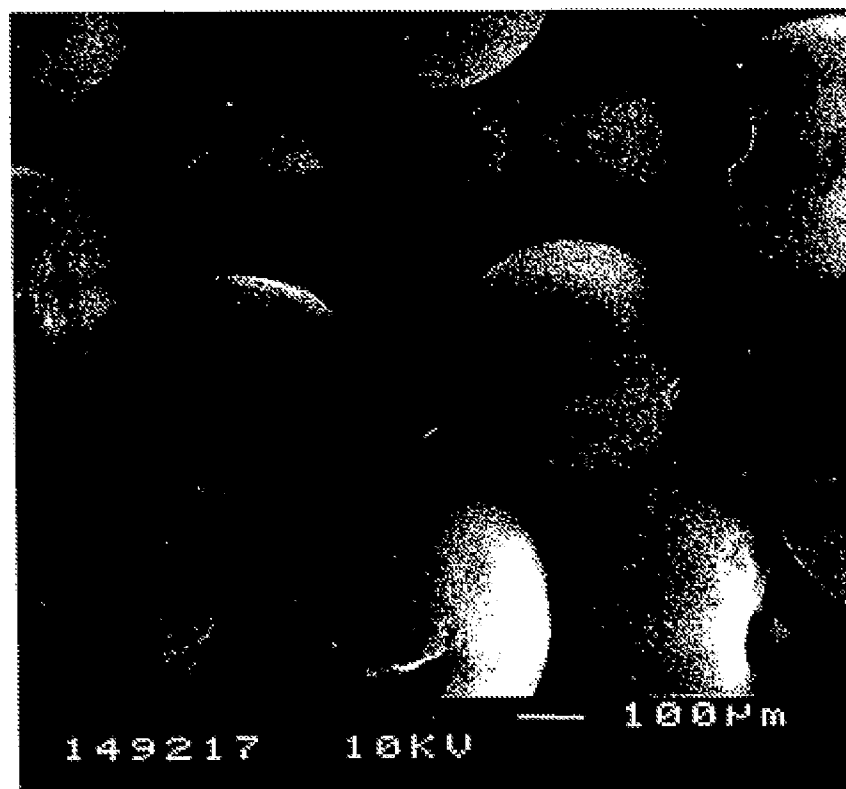
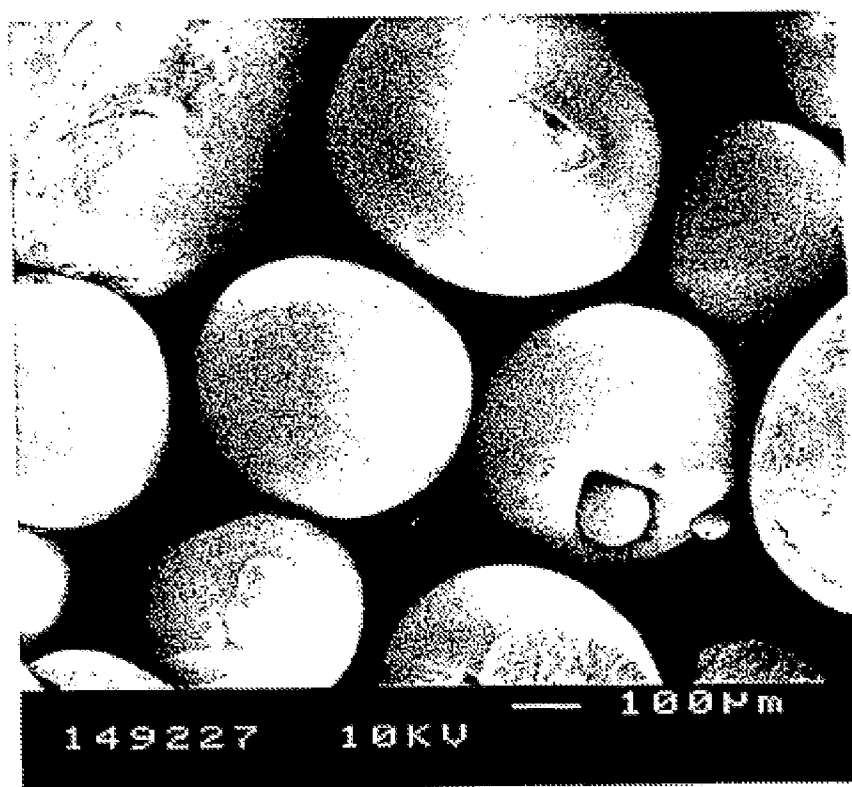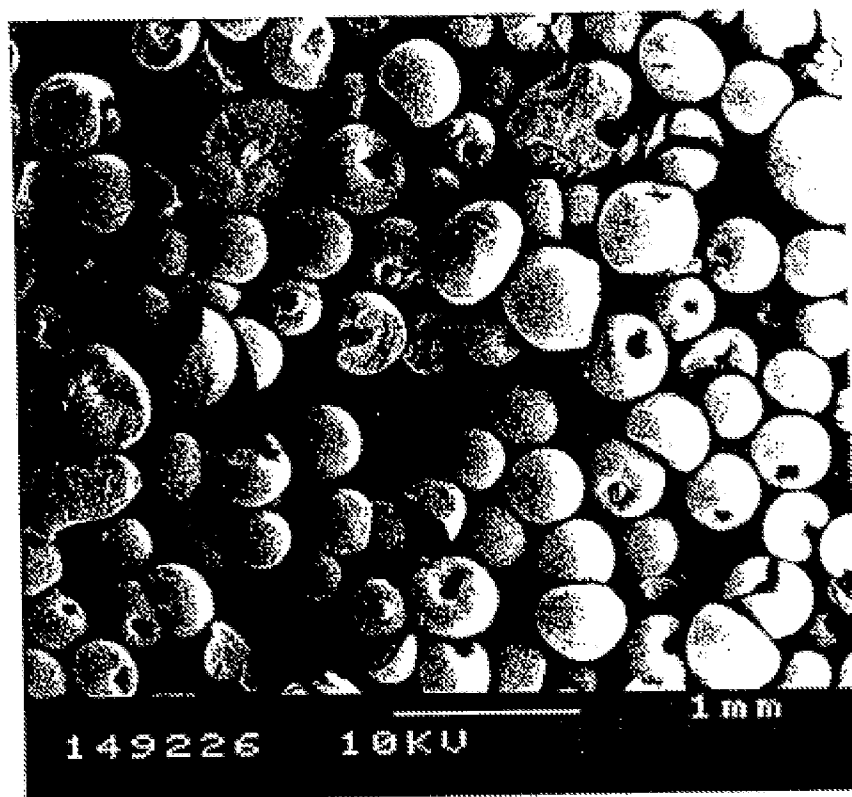Figure 2 Grain distribution function of the FSD – test products, compared to Sipernat 22

Key: a) Alpine oversize material[%]; b) test FSD 1; c) test FSD 3; d) Sipernat 22f

Figur 3

Figur 4

149226　10KV　　　　1mm

149227　10KV　　　100µm

Figur 5

149231   10KU   1 m m

149232   10KU   100 μm